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STUDY OF DOPANTS FOR RADIATION-RESISTANT SILICON

QUARTERLY REPORT

OCTOBER 1969

Letter Contract No. 952523

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NORTHROP CORPORATE LABORATORIES

3401 West Broadway
Hawthorne, California 90250

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NOTICE

This report contains information prepared by Northrop Corporate Laboratories under JPL subcontract. Its content is not necessarily endorsed by the Jet Propulsion Laboratory, California Institute of Technology, or the National Aeronautics and Space Administration.

NEW TECHNOLOGY

All technological development to date are reported herein. They are considered to be unreportable under the instructions of NHB 2170.2 dated October 1966.

ABSTRACT

During this quarter, a number of lifetime samples were prepared and characterized using the existing supply of Al- and B-doped material. Highly uniform samples were prepared by diffusing lithium into blanks made of ~ 160 ohm cm P-doped material, to be used to evaluate further the effect of lithium on the radiation response of silicon. In addition, four Al-doped crystals representing different dopant concentrations (5 and 10 ohm cm) and different crystal growth techniques (Czochralski and float-zone) have been ordered from each of two different suppliers for a detailed study of the effect of this impurity on lifetime degradation in bulk silicon.

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INTRODUCTION

This report describes activities during the past three months in a study of dopants for radiation-resistant silicon. The results of past studies (JPL contract 952256) and investigations of neutron effects indicate that under some circumstances aluminum-doped silicon is radiation resistant. For this reason particular emphasis is being given to this material. Efforts are also being made to produce high quality large bulk samples of lithium-doped silicon to provide quantitative information concerning the interactions between lithium and radiation-induced defects in neutral bulk material. The aluminum-doped crystals employed in this study are custom grown, by outside suppliers. Lithium-doped samples are prepared in our laboratory by diffusing this impurity into bulk samples made from ~ 160 ohm cm phosphorus-doped material obtained from an outside supplier.

TECHNICAL DISCUSSION

SAMPLE PREPARATION

Eight lifetime samples were prepared from remaining portions of three Al-doped crystals which were purchased under the previous JPL contract. All three crystals were grown using float-zone techniques and were known from previous measurements to have reasonably long initial lifetimes. These samples will be irradiated with similar samples which will be prepared from new crystals now on order.

In addition to these Al-doped samples, four samples were prepared from two 10 ohm cm B-doped crystals. These samples are to be used as controls for determining the relative radiation sensitivity of material containing non-conventional dopants in future experiments.

Lifetime measurements were performed on all of these samples as a function of temperature in the temperature range from approximately +200 °C to -65 °C [$2.1 \leq 10^3/T(^{\circ}\text{K})^{-1} \leq 4.8$]. These measurements are necessary for an understanding of the recombination process in these materials and for revealing the nature of radiation-induced recombination centers.

LITHIUM DIFFUSION STUDIES

Significant improvements in both the uniformity and lifetime of lithium diffused samples have been obtained by modifications in the diffusion cycle. Those improvements are the result of a number of experiments which were performed to determine the optimum techniques for producing uniform distributions and desirable concentrations of lithium in bulk lifetime samples. The source of lithium used in all of these experiments was a solution of lithium-aluminum hydride in ether. Bulk lifetime samples measuring approximately 7 x 7 x 30 mm were cut from a crystal of P-doped silicon grown by Texas Instruments using the Lopex technique (modified float-zone). The initial room-temperature lifetime of this material was

approximately 800 microseconds and the resistivity was approximately 160 ohm cm. Freshly etched samples of this material were immersed in the lithium-aluminum hydride solution and were then heated in vacuum at various temperatures and for various periods of time to diffuse the lithium. Following the diffusion cycle, the samples were lapped and etched to remove any excess lithium from or near the surface and were then reheated in vacuum to distribute the lithium more uniformly through the sample.

In most of the earlier experiments, the diffusions were performed at 425°C or 450°C and for minimum periods of 30 minutes. These treatments produced samples with lower resistivities than desired and it was not possible to distribute the apparently high concentration of lithium uniformly through the samples by subsequent long distribution cycles. The improvement in the quality of samples produced in the more recent experiments has been due to the use of diffusion times of from approximately 5 to 15 minutes and a reduction of the diffusion temperature to 400°C. For convenience, all of the samples were heated for approximately 24 hours to distribute the lithium. However, this time can probably be reduced considerably without seriously affecting the quality of the samples.

Some of the properties of the more recently prepared samples are illustrated in Figures 1 through 5. Figures 1 and 2 are potential profiles which illustrate the uniformity of a sample before and after the lithium diffusion process. Potential profiles are obtained for all of the samples employed in this work. They are obtained by passing a constant current through the sample and measuring the voltage as a function of position along the length. The voltage varies linearly with position in uniform material (constant resistivity), but gradients in the dopant concentration produce non-linear profiles. Thus these figures demonstrate the uniformity of both starting and doped material. In addition to providing information regarding the sample uniformity, these profiles are used to determine the sample resistivity and to evaluate the quality of the electrical contacts. Poor

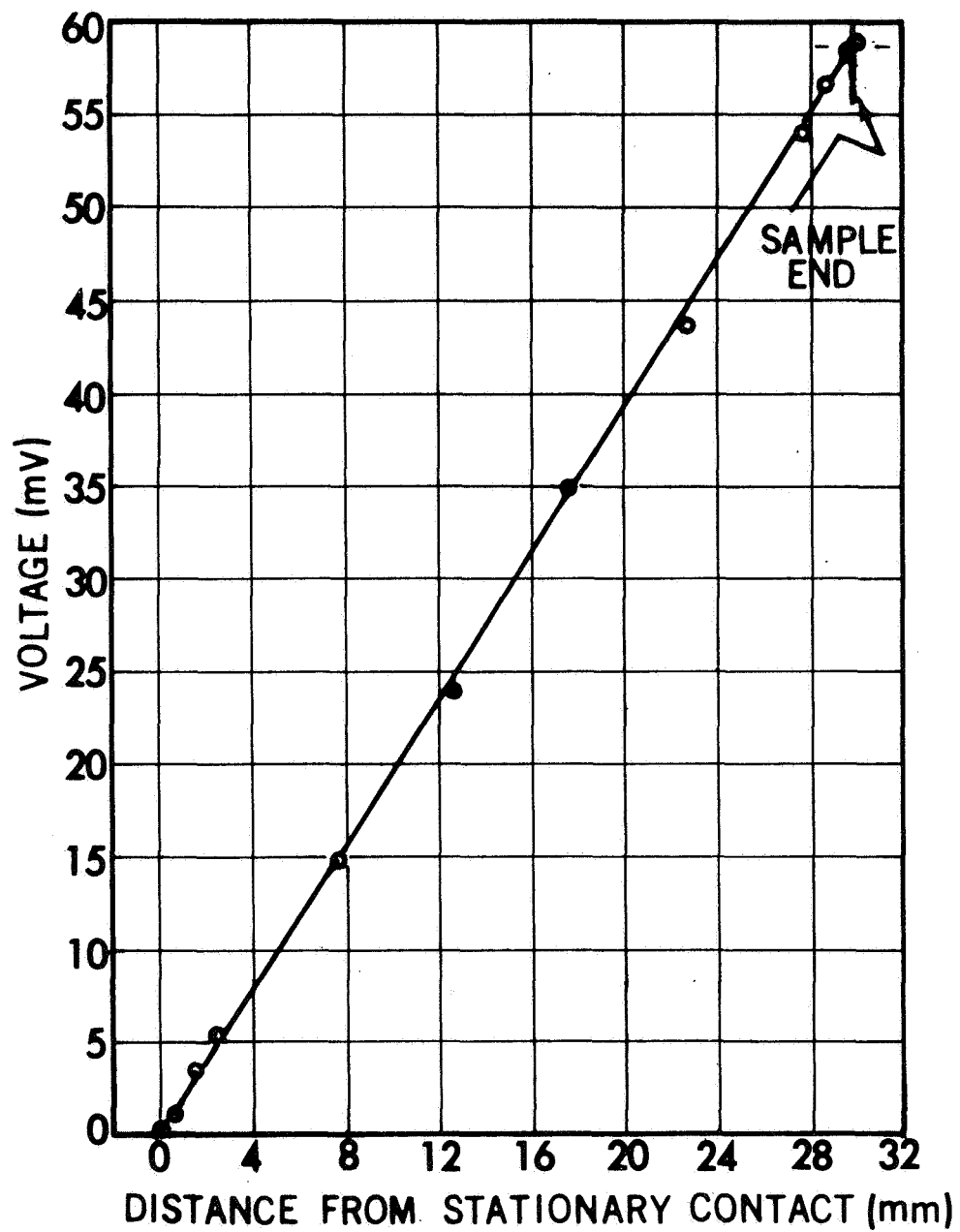


Figure 1 Potential profile of a typical lifetime sample made from TLP 160 crystal as received.

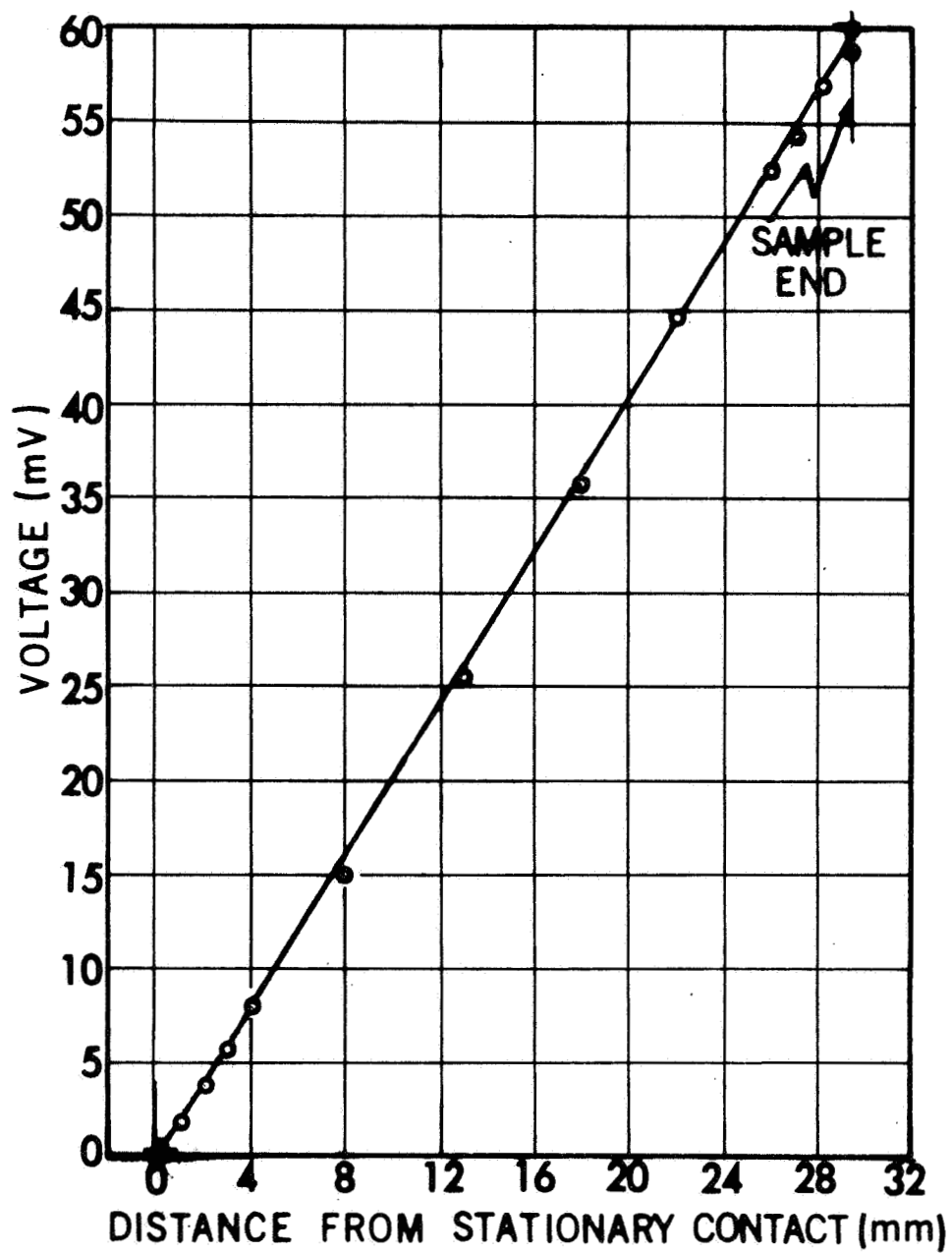


Figure 2 Potential profile of a 12 ohm cm lifetime sample prepared by diffusing Li into TLP 160 material.

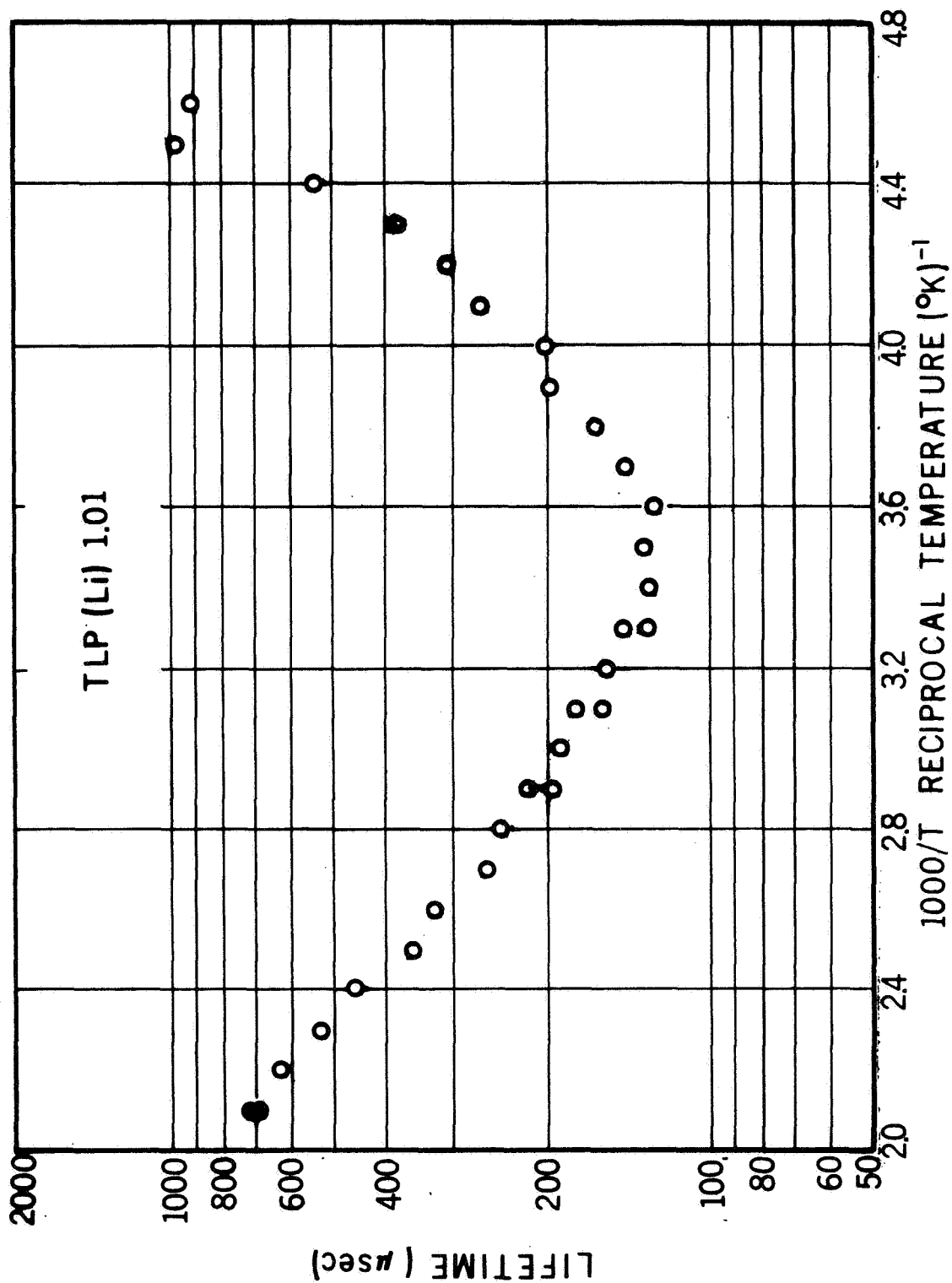


Figure 3 Temperature dependence of lifetime in ~ 1 ohm cm Li-doped silicon.

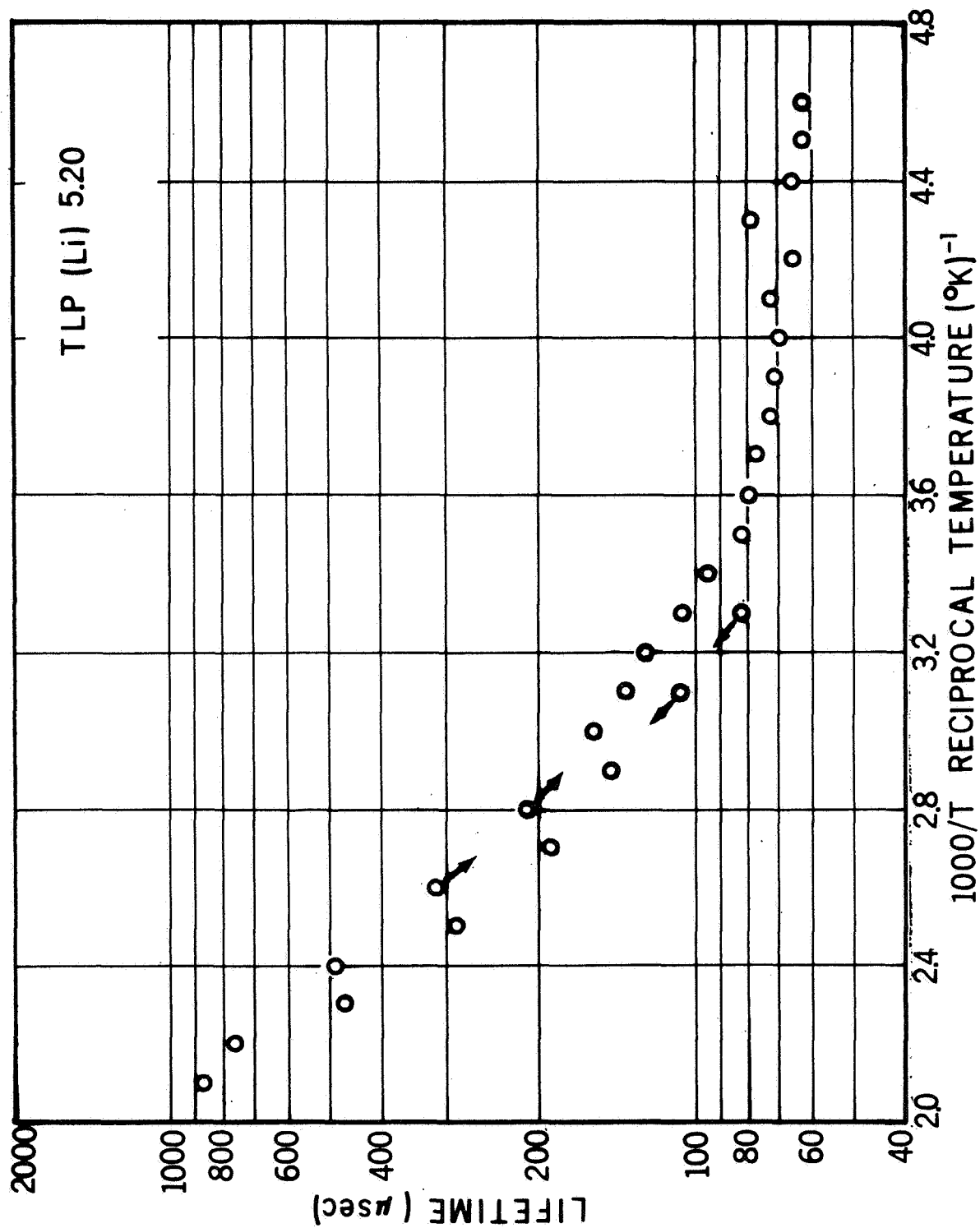


Figure 4 Temperature dependence of lifetime in ~ 5 ohm cm Li-doped silicon.

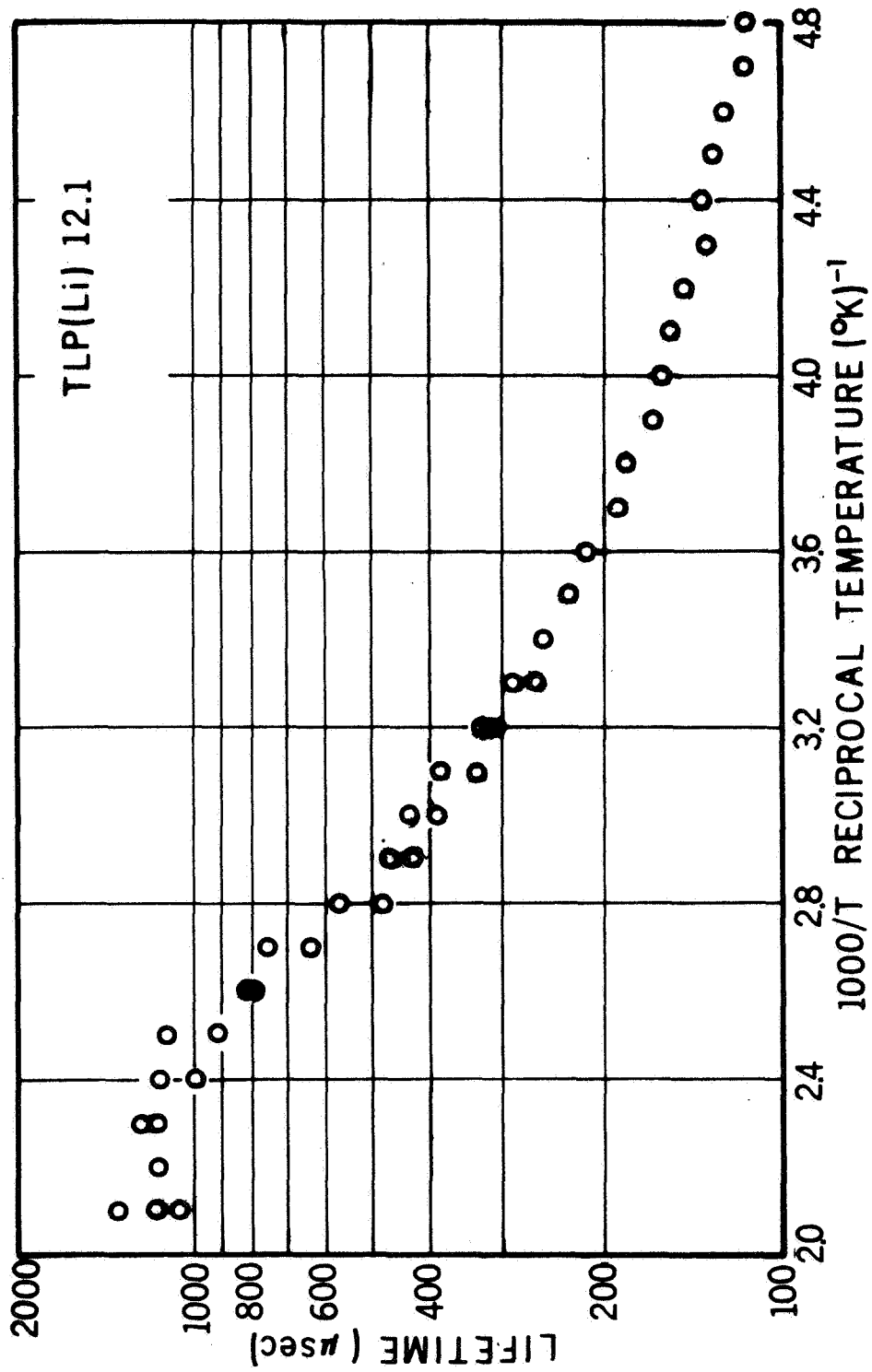


Figure 5 Temperature dependence of lifetime in ~ 12 ohm cm Li-doped silicon.

contacts produce relatively large voltage drops near the end of the sample and result in discontinuities in the potential profile.

Figures 3 through 5 illustrate the temperature dependence of the lifetime in samples containing different concentrations of lithium. The minimum in the curve of Figure 3 is tentatively attributed to trapping produced by a center which becomes dominant slightly below room temperature [$10^3/T(^{\circ}\text{K})^{-1} \approx 3.3$]. A similar minimum was observed in all of the material prepared to date which had a resistivity lower than approximately 5 ohm cm. Note that the samples indicated in Figures 4 and 5 do not exhibit the minimum although the lifetime of the 5 ohm cm sample of Figure 4 remains nearly constant at lower temperatures. Note also that the lifetime of this sample increased after it was heated to $\sim 200^{\circ}\text{C}$ [$10^3/T(^{\circ}\text{K})^{-1} = 2.1$] in performing the temperature dependence measurements.

Figure 5 illustrates the lifetime behavior of the sample whose potential profile is shown in Figure 2. This sample was prepared using a diffusion cycle of approximately 8 minutes at 400°C and a distribution cycle of 24 hours at the same temperature. The relatively long lifetime and absence of trapping effects indicate that the sample should prove particularly useful for future degradation studies.

MATERIAL PROCUREMENT

Orders were placed with both the General Electric Company and Texas Instruments, Inc. for Al-doped crystals grown by both the Czochralski (pulled) and float-zone techniques and having Al concentrations corresponding to nominal resistivities of 5 and 10 ohm cm. In addition, one or more B-doped crystals in this resistivity range were ordered from each manufacturer to provide control samples for evaluating the radiation response of material containing non-standard dopants.

In our previous studies of radiation effects in Al-doped material it was not possible to evaluate the influence of oxygen on the observed radiation

response because the initial lifetime of pulled material was too low to permit accurate measurement after irradiation. However, both suppliers indicated that they can provide pulled crystals with lifetimes greater than 20 microseconds and, if so, the new crystals should permit a valid evaluation of the effect of oxygen on the radiation response of this material.

Unfortunately, heavy production schedules and other factors beyond external control at both General Electric and Texas Instruments have resulted in delays of up to seven weeks in the delivery of custom-grown crystals. Because of these delays and the additional time required to obtain formal price quotes from the manufacturers, none of the new crystals have been delivered to date. General Electric has grown some of the crystals, however, and delivery is expected shortly following auxiliary measurements of the resistivity gradient, oxygen concentration and approximate bulk lifetime to insure that our specifications have been met. None of the pulled crystals grown to date has been acceptable because of large resistivity gradients. These gradients are due to the extremely small segregation coefficient of Al in Si (0.002) and may be unavoidable in pulled crystals since the Al tends to remain in the molten silicon. A low segregation coefficient is beneficial in producing crystals with uniform dopant concentrations using float-zone processes, however. Because of this effect, all of the float-zone crystals which we have employed in previous experiments had very small resistivity gradients.

CONCLUSIONS

Suitable samples for lifetime degradation studies can be produced by diffusing lithium into bulk silicon having a sufficiently high initial lifetime. However, to avoid excessive trapping effects, and to permit uniform distribution of the lithium, the lithium concentration should be kept as small as practical. Further experiments are expected to reveal that the diffusion temperature and/or the 24 hour distribution time presently employed can be reduced without adversely affecting the sample quality.

FUTURE PLANS

Lifetime samples will be prepared and characterized from each of the new Al-doped crystals as soon as they are delivered. Additional Li-doped samples will be prepared by diffusion using existing techniques. Attempts will also be made to produce suitable samples by diffusing at lower temperatures and employing shorter distribution times.